

STUDY TITLE

Acute Toxicity Evaluation of Step I Pipe Discharge (WS006/WS007) from US Steel-MinnTac, Mountain Iron, Minnesota

DATA STANDARD

“Methods for Measuring the Acute Toxicity of Effluents and Receiving Waters to Freshwater and Marine Organisms,” 2002, Fifth Edition. EPA/821/R/02/012. U.S. Environmental Protection Agency, Washington, DC.

STUDY DATES

November 5-7, 2015

STUDY SUBMITTED

November, 2015

TESTING FACILITY

Pace Analytical  
4730 Oneota Street  
Duluth, MN 55807

Tel. No. (218) 727-6380

PROJECT IDENTIFICATION NUMBERS

Pace Project # 088594

NPDES Permit No. MN0057207

## 1.0 EXECUTIVE SUMMARY AND RESULTS

MinnTac Step I Pipe Discharge (WS006/WS007), Mountain Iron, MN was evaluated for acute toxicity to fathead minnows (*Pimephales promelas*) during November 5-7, 2015. McNiven Creek Water (SW002) was used as the dilution and primary control water for the test. Moderately hard reconstituted water (MHRW) was used as the laboratory reference control water for the tests. The table below summarizes the toxicity results of the tests.

***Fathead Minnow***

48 Hour LC<sub>50</sub>: >100%

(95% C.I. Not Calculable)

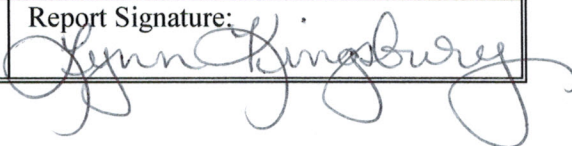
Survival NOEC: 100%

Survival LOEC: --

Toxicity Units (TUa): <1.00

*The chemistry determinations for the final effluent samples are attached at the end of this report. The data package and statistical analysis for the test battery is also attached.*

## 2.0 GENERAL INFORMATION

Data Standard	EPA/821/R/02/012	
Testing Facility	Pace Analytical, 4730 Oneota Street, Duluth, MN 55807, Tel. No. (218) 727-6380	
Project Director	Joseph Dierkes	
Test Dates	November 5-7, 2015	
Report Approval	Lynn Kingsbury, Lab Analyst III-Bioassay	Report Signature: 

## 3.0 OBJECTIVE

To determine the median lethal concentration (LC<sub>50</sub>), no- & lowest- observed effect concentrations (NOEC & LOEC) based on survival of MinnTac Step I Pipe Discharge (WS006/WS007) for fathead minnows.

## 4.0 REFERENCES

American Public Health Association (APHA). 1998. 20<sup>th</sup> Ed. of Standard Methods for the Examination of Water and Wastewater. APHA, Washington, D.C.

USEPA. 2002. Methods for Measuring the Acute Toxicity of Effluents and Receiving Waters to Freshwater and Marine Organisms. Fifth Edition. EPA/821/R/02/012. U.S. Environmental Protection Agency, Washington, DC.

MPCA Acute Toxicity Test Conditions for Static Renewal Whole Effluent Tests. Minnesota Pollution Control Agency.

West Inc. & University of Wyoming. TOXSTAT Software Package Version 3.5.

## 5.0 TEST METHODS

**Test Concentrations:** 0 (dilution water control), and 100%.

**Test Organisms:** Fathead minnow (*Pimephales promelas*) 10 days old.

**Test Chambers:** Fathead minnow: 800 mL glass beakers.

**Incubation:** *Duration:* 48 hours. *Renewals:* None

**Observations:** Survival was recorded each day. The test was terminated following 48 hours of exposure.

**Endpoint Calculations:** LC<sub>50</sub> USEPA Statistical Programs. NOEC and LOEC: TOXSTAT statistical software, Version 3.5, West, Inc., and University of Wyoming.

## 6.0 DEVIATIONS FROM REFERENCED METHOD

To the best of our knowledge, no deviations from the referenced method (EPA-821-R-02-012) occurred for the study.

### Cumulative Percentage of Surviving Organisms for Selected Test Species Exposed to MinnTac Step I Pipe Discharge (WS006/WS007) Tested November 5-7, 2015

Effluent Concentration (%)	Test Interval	
	24 hour	48 hour
Fathead Minnow		
0 (MHRW) <sup>a</sup>	100 <sup>c</sup> (20/20) <sup>d</sup>	95 (19/20)
0 (RW) <sup>b</sup>	100 (20/20)	100 (20/20)
100	100 (20/20)	100 (20/20)

<sup>a</sup> Secondary Control was Moderately Hard Reconstituted Water (MHRW)

<sup>b</sup> Primary Control Water was McNiven Creek Water (SW002)

<sup>c</sup> Cumulative Percentage Survival

<sup>d</sup> # Alive/# Exposed





**Minnesota Pollution  
Control Agency**

520 Lafayette Road North  
St. Paul, MN 55155-4194

# Acute Whole Effluent Toxicity Test Report

## NPDES Permit Program

Doc Type: Effluent Limit Standards Review

Please read all instructions carefully before completing each section of the report. **Instructions are found on Page 5.**

**Address the completed, signed report to:** Attn: Water Quality Submittals  
Minnesota Pollution Control Agency  
520 Lafayette Road North  
St. Paul, Minnesota 55155-4194

### Facility Information

Permit No.: MN0057207 Station ID: WS006/WS007  
Name of permittee: US Steel Minntac

### Test Information

Test start date (mo/day/year): 11/5/15 Test end date (mo/day/year): 11/7/15

or

☐ Check if **no discharge occurred** and leave the rest of the form blank.

Report completion date (mo/day/year): 11/12/15

WET test type (check one): ☒ Initial ☐ Repeat #1 ☐ Repeat #2 ☐ TAC Repeat

For repeats, test start date of initial WET test (mo/day/year): \_\_\_\_\_

Dilution water (check one): ☐ Lab ☒ Receiving water

If receiving water, indicate name: McNiven Creek Water (SW002)

Test lab name: Pace Analytical Phone: (218) 727-6380

Mailing address: 4730 Oneota Street

City: Duluth State: Minnesota Zip code: 55807

Lab representative name: Joseph Dierkes E-mail: Joe.dierkes@pacelabs.com

Facility representative name: Thomas Moe E-mail: tmoe@uss.com



# Sample condition upon test initiation

Sample	Collection date	Receipt date	First use date	pH (SU)	Conductivity (umhos/cm)	TRC (mg/L)	Temp °C Arrival at lab	Temp °C upon test initiation	Total Ammonia (mg/L)	Alkalinity (mg/L)	Hardness (mg/L)
Receiving Water 1	11/4/15	11/5/15	11/5/15	6.7	96	<0.02	6.1	25	<0.06	20	36
Receiving Water 2*											
Receiving Water 3*											
Receiving Water 4*											
Effluent 1	11/4/15	11/5/15	11/5/15	8.1	2180	<0.02	11.6	25	1.60	206	842
Effluent 2*											
Effluent 3*											
Effluent 4*											

\*If applicable umhos/cm = micromhos per centimeter mg/L = milligrams per Liter (mg/L)

Does facility have chlorine limit? (check one) ☐ Yes ☒ No

Was sample dechlorinated at lab? (check each one that applies)

☐ Effluent 1 ☐ Effluent 2 ☐ Effluent 3 ☐ Effluent 4

Effluent filtered? (check one) ☐ Yes ☒ No If yes, state mesh size: \_\_\_\_\_

Effluent sample type (check one type for each sample):

Effluent 1: ☐ 24 hr composite ☒ Grab ☐ Grab-composite – Enter number of grabs: \_\_\_\_\_

Effluent 2: ☐ 24 hr composite ☐ Grab ☐ Grab-composite – Enter number of grabs: \_\_\_\_\_

Effluent 3: ☐ 24 hr composite ☐ Grab ☐ Grab-composite – Enter number of grabs: \_\_\_\_\_

Effluent 4: ☐ 24 hr composite ☐ Grab ☐ Grab-composite – Enter number of grabs: \_\_\_\_\_

### Summary of data for Fathead Minnows – Percent survival per concentration

Day	0 <sub>1</sub> (diluent)	0 <sub>2</sub> (if used)	%	%	%	%	100%
1	100	100					100
2	100	95					100
3							
4							

### Summary of data for *Ceriodaphnia dubia* – Percent survival per concentration

Day	0 <sub>1</sub> (diluent)	0 <sub>2</sub> (if used)	%	%	%	%	100%
1							
2							

### Summary of data for *Daphnia magna* – Percent survival per concentration

Day	0 <sub>1</sub> (diluent)	0 <sub>2</sub> (if used)	%	%	%	%	100%
1							
2							

### Summary of results – Acute values

Species	LC <sub>50</sub>	TU <sub>a</sub> (acute toxic units)
Fathead Minnow (96 hour)	>100%	<1.00
<i>Ceriodaphnia dubia</i> (48 hour)		
<i>Daphnia magna</i> (48 hour)		

### Test Acceptability Criteria (TAC)

QA/QC Criteria	Criteria met for Acute Toxicity
Fathead Minnow – Age range <24 hours at test start	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No
<i>Ceriodaphnia dubia</i> – Age range <24 hours at test start	<input type="checkbox"/> Yes <input type="checkbox"/> No
<i>Daphnia magna</i> – Age range <24 hours at test start	<input type="checkbox"/> Yes <input type="checkbox"/> No
Test started within 36 hours of collection of sample	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No
90% or greater survival of all control organisms (if any species results fail, check No)	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No

If reference toxicant other than NaCl used, please indicate: \_\_\_\_\_

### Test deviation comments

Describe any deviation from test methods: Per permit requirements only 48 hour fathead minnow test performed with no dilution.

(For example: pH-controlled test amount of CO<sub>2</sub>, reduced DO levels in test leading to aeration, sample exceeded holding time.)

## Certifications

Form shall be signed by the laboratory here certifying the results:

Lab representative (print): Lynn Kingsbury Phone: (218) 727-6380

Signature: 

Date: 11/12/15

Form shall be signed by the permittee here in accordance with Minn. R. 7001.0540 Certification of Permit Applications and Reports

*I certify under penalty of law that this document and all attachments were prepared under my direction or supervision in accordance with a system designed to assure that qualified personnel properly gather and evaluate the information submitted. Based on my inquiry of the person or persons who manage the system or those persons directly responsible for gathering the information, the information submitted is, to the best of my knowledge and belief, true, accurate, and complete. I am aware that there are significant penalties for submitting false information, including the possibility of fine and imprisonment.*

Permittee representative (print): Tom Moe Phone: (218) 749-7485

Signature: \_\_\_\_\_ Date: \_\_\_\_\_

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### Attachments provided:

Data package, Narrative report, Reference Toxicant Chart (Reference test met requirements).



# Whole Effluent Toxicity Test Instruction Sheet

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This instruction sheet is provided to aid in filling out the Acute or Chronic Whole Effluent Toxicity (WET) Test report forms as required by your National Pollutant Discharge Elimination System (NPDES) Permit. If your permit requires acute WET testing, there will be one form to fill out. If your permit requires chronic WET testing, there will be two forms to fill out.

## Section 1. Facility Information

Fill out your NPDES permit number, discharge outfall station identification (ID) number, and your Permittee name as it appears on your NPDES permit.

## Section 2. Test Information

**Test Start Date/Test End Date.** Fill out the date the test began (month/day/year), the date the test finished (month/day/year)

**No discharge.** This box should be checked if no discharge occurred during the WET monitoring period. If no discharge occurred, the rest of the form is left blank, and should be signed, dated, and submitted by the required date.

**Report Completion Date.** Fill out the completion date, which is the date the report is sent from the testing laboratory as indicated on the test report (month/day/year).

**WET test type.** Select the appropriate form to fill out. This will be either the acute test form, or both the Fathead chronic minnow form and the *Ceriodaphnia* chronic form. Check the appropriate test represented by this report; either Initial, Repeat#1, Repeat#2, or TAC Repeat

**Dilution water.** Check either lab or receiving water, whichever was used to dilute the effluent sample to create the test series concentrations. If receiving water is used as the diluents, supply the name of that receiving water.

**Test Lab Name, etc.** Provide the name, phone number, address, lab representative name, and e-mail address.

**Facility representative name.** Provide the name of the discharging facility's representative and e-mail address.

**Sample condition table.** The laboratory conducting the tests must fill in the sample information and dates associated with the samples received. The information is typically collected for Chain of Custody forms and for initial sampling upon initiating a test. Provide the date the sample was collected, receipt date at the lab, date of first use for testing, pH, specific conductivity, total residual chlorine (TRC) analysis, temperature upon arrival at the lab and temperature at test initiation, total ammonia analysis, total alkalinity, and total hardness. Sample data will be provided for each sample received and as specified by the applicable acute or chronic manuals, or by the permit. Up to four samples each may be received for the receiving water and effluent for acute tests. Up to three samples may be received for chronic tests for the receiving water. Fill in any data that is below quantification levels with a less than sign and that actual quantification level (e.g. <40 ug/l) – **do not report ND**.

**Does facility have a chlorine (TRC) permit limit?** Check either Yes or No.

**Was sample dechlorinated at the lab?** Check each sample that was dechlorinated at the lab.

**Effluent filtered?** Check either Yes or No, and state the mesh size used for filtration.

**Effluent sample type.** For each sample collected check or check whether the sample was a twenty-four hour composite, a grab, or a grab composite with the number of grabs taken to comprise the composite sample.

**Summary of data for each species for acute or chronic tests, as applicable in your permit.** For each species in an acute test summarize the cumulative survival data over all replicates for each treatment level for each day of the test. Fill in the dilution series expressed as percent effluent as specified by the permit. Reaffirm which sample of either receiving water or lab water was used as the diluent (O<sub>1</sub>) based on your answer in section 2 *Test Information – Dilution water*.

For a Fathead minnow chronic test summarize the cumulative 96 hour survival data over all replicates, the cumulative mean 7-day biomass data, and the 7-day cumulative mean percent survival data for each treatment level for the test. Fill in the dilution series expressed as percent effluent as specified by the permit. Reaffirm which sample of either receiving water or lab water was used as the diluent (O<sub>1</sub>) based on your answer in section 2 *Test Information – Dilution water*.

For a *Ceriodaphnia* chronic test summarize the cumulative 48 hour survival data over all replicates, the cumulative mean 7-day reproduction/female data, and the 7-day cumulative mean percent survival data for each treatment level for the test. Fill in the dilution series expressed as percent effluent as specified by the permit. Reaffirm which sample of either receiving water or lab water was used as the diluent (O<sub>1</sub>) based on your answer in section 2 *Test Information – Dilution water*.

**Summary of results table for each species for acute or chronic tests, as applicable.** For acute tests provide the appropriate 48 or 96 hour LC50 values for each species and the calculated toxic unit values (TUa for acute or TUC for chronic tests).

For Fathead minnow chronic tests provide the calculated IC<sub>25</sub> for larval survival and growth at the end of the test as well as the critical No Observed Effect Concentration (NOEC) and Lowest Observed Effect Concentration (LOEC). Also provide the chronic toxic units (TUC) calculated as the inverse of the IC<sub>25</sub> value.

For *Ceriodaphnia* chronic tests provide the calculated IC<sub>25</sub> for survival and reproduction at the end of the test as well as the No Observed Effect Concentration (NOEC) and Lowest Observed Effect Concentration (LOEC). Also provide the chronic toxic units (TUC) calculated as the inverse of the IC<sub>25</sub> value.

**Test acceptability criteria.** For acute tests check the corresponding boxes either yes or no for each of the Quality Assurance/Quality Control (QA/QC) criteria.

For chronic tests check the corresponding boxes either yes or no for each of the QA/QC criteria. If there is deviation from the specified methods, please describe the change and reason for the deviation. PMSD is the percent minimum significant difference from hypothesis testing that characterizes overall test variability. The temperature variation of  $\pm 1^{\circ}$  C is for individual measured test chambers over a test.

**Deviations from test methods.** If there is any deviation from the specified methods, please describe the change and the reason for the deviation. This is a narrative comment area to describe any unusual circumstances in conducting any of these tests.

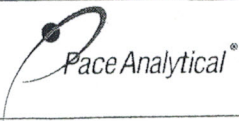
**Certifications.** Provide the laboratory representative name, signature, phone number and date for the tests completed. Also provide the permittee representative name, signature, phone number and date for the tests completed. The Permittee is responsible for forwarding these results to the Minnesota Pollution Control Agency.

**Attachments.** Attach any narrative regarding results or SOP, bench sheets for analytical and biological data, chain of custody forms, reference test results for tested species (tables and/or charts over the past year), and toxicity calculation outputs.







	Document Name:	Document Revised: 09Sep2015
	Sample Condition Upon Receipt Form	Page 1 of 1
	Document No.: F-DUL-C-001-Rev.00	Issuing Authority: Pace Virginia, Minnesota Quality Office

<b>Sample Condition Upon Receipt</b>	Client Name:	Project #:
	<div style="display: flex; justify-content: space-around;"> <div style="border: 1px solid black; padding: 5px;">088594-1 11-05-15</div> <div style="border: 1px solid black; padding: 5px;">088594-2 11-05-15</div> </div>	
Courier:	<input checked="" type="checkbox"/> Fed Ex <input type="checkbox"/> UPS <input type="checkbox"/> USPS <input type="checkbox"/> Client <input type="checkbox"/> Commercial <input type="checkbox"/> Pace <input type="checkbox"/> Other:	
Tracking Number:	(0215) 8057 8288 3777 (0215) 8057 8288 3766	
Custody Seal on Cooler/Box Present?	<input type="checkbox"/> Yes <input checked="" type="checkbox"/> No	Seals Intact? <input type="checkbox"/> Yes <input type="checkbox"/> No
Packing Material:	<input type="checkbox"/> Bubble Wrap <input type="checkbox"/> Bubble Bags <input checked="" type="checkbox"/> None <input type="checkbox"/> Other:	Temp Blank? <input type="checkbox"/> Yes <input checked="" type="checkbox"/> No
Thermometer Used:	<input checked="" type="checkbox"/> B00051	Type of Ice: <input checked="" type="checkbox"/> Wet <input type="checkbox"/> Blue <input type="checkbox"/> None <input checked="" type="checkbox"/> Samples on ice, cooling process has begun
Cooler Temp Read °C:	11.0 5.5	Cooler Temp Corrected °C: 11.4 6.1
Temp should be above freezing to 6°C	Correction Factor: +0.4	Biological Tissue Frozen? <input type="checkbox"/> Yes <input type="checkbox"/> No <input checked="" type="checkbox"/> NA
		Date and Initials of Person Examining Contents: 11-5-15 CA

			Comments:
Chain of Custody Present?	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	1.	
Chain of Custody Filled Out?	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	2.	
Chain of Custody Relinquished?	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	3.	
Sampler Name and Signature on COC?	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	4.	
Samples Arrived within Hold Time?	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	5.	
Short Hold Time Analysis (<72 hr)?	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	6.	pH, res. chl.
Rush Turn Around Time Requested?	<input type="checkbox"/> Yes <input checked="" type="checkbox"/> No <input type="checkbox"/> N/A	7.	
Sufficient Volume?	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	8.	
C Containers Used?	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	9.	
-Pace Containers Used?	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A		
Containers Intact?	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	10.	
Filtered Volume Received for Dissolved Tests?	<input type="checkbox"/> Yes <input type="checkbox"/> No <input checked="" type="checkbox"/> N/A	11.	Note if sediment is visible in the dissolved containers.
Sample Labels Match COC?	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	12.	
-Includes Date/Time/ID/Analysis Matrix: WT			
All containers needing acid/base preservation have been checked?	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	13.	<input checked="" type="checkbox"/> HNO <sub>3</sub> <input checked="" type="checkbox"/> H <sub>2</sub> SO <sub>4</sub> <input type="checkbox"/> NaOH <input type="checkbox"/> HCl Sample # 088594-1 & -2 pH = 2 Initials when Completed: CA Lot # of added preservatives: 10/215-2A 10/215-4CFX
All containers needing preservation are found to be in compliance with EPA recommendation?	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A		
HNO <sub>3</sub> , H <sub>2</sub> SO <sub>4</sub> , HCl <2; NaOH >9 Sulfide, NaOH >12 Cyanide)	<input type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A		
Exceptions: VOA, Coliform, TOC, Oil and Grease, DRO/8015 (water) DOC	<input type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A		
Exceptions: VOA, Coliform, TOC, Oil and Grease, DRO/8015 (water) DOC	<input type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	14.	
Trip Blank Present?	<input type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	15.	
Trip Blank Custody Seals Present?	<input type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A		
Pace Trip Blank Lot # (if purchased):			

<b>CLIENT NOTIFICATION/RESOLUTION</b>	Field Data Required? <input type="checkbox"/> Yes <input type="checkbox"/> No
Person Contacted: _____	Date/Time: _____
Comments/Resolution: _____	

FECAL WAIVER ON FILE    Y    N	TEMPERATURE WAIVER ON FILE    Y    N
Project Manager Review: _____	Date: _____
Note: Whenever there is a discrepancy affecting North Carolina compliance samples, a copy of this form will be sent to the North Carolina DEHNR Certification Office (i.e. out of hold, incorrect preservative, out of temp, incorrect containers)	



Pace Analytical

4730 Oneota Street Duluth MN 55807 Telephone: (218)727-6380 Fax: (218)727-3049

### Laboratory Report

Project Number: 088594  
COC Number: 088594  
Date Received: 11/5/2015  
Report Date: 11/11/2015  
Report Number: 136386

Client: TOM MOE  
U.S. STEEL MINNTAC  
MINNESOTA ORE OPERATIONS  
P O BOX 417  
MT IRON MN 55768

Sample ID: WS006 / WS007  
Pace Analytical Project Number: 088594-1

Parameter:	Results:	Temperature upon arrival (°C):		Analysis Date/Time:	Sample Date:	Method:	DF:	LOD:	LOQ:	Matrix:	QC Comments:
		Units:									
Alkalinity (as CaCO <sub>3</sub> )	206	mg/L	11/10/2015	13:04	11/4/2015	SM 2320 B-97	1	1	3	Aqueous	
Nitrogen, Ammonia	1.60	mg/L	11/9/2015	12:12	11.6	SM 4500-NH3 D-97	1	0.06	0.2		
pH - Lab	8.1	SU	11/5/2015	15:40		SM 4500-H+B-96	1				
Residual Chlorine - Lab	<	mg/L	11/5/2015	10:30		SM 4500-Cl D-93	1	0.02	0.07		
Specific Conductance	2180	µmhos/cm	11/10/2015	13:57		EPA 120.1	1	1	10		
Hardness, Total (as CaCO <sub>3</sub> )	842	mg/L	11/10/2015	9:30		USGS I-1338-85	2	4	10		



Pace Analytical

4730 Oneota Street Duluth MN 55807

Telephone: (218)727-6380

Fax: (218)727-3049

### Laboratory Report

Project Number: 088594  
COC Number: 088594  
Date Received: 11/5/2015  
Report Date: 11/11/2015  
Report Number: 136386

Sample ID:	SW002	Sample Date:	11/4/2015	Sample Time:	9:20	Matrix:	Aqueous
Pace Analytical Project Number:	088594-2	Temperature upon arrival (°C):	6.1				
<u>Parameter:</u>		<u>Results:</u>	<u>Units:</u>	<u>Analysis Date/Time:</u>	<u>Method:</u>	<u>DF:</u>	<u>LOD:</u> <u>LOQ:</u> <u>QC Comments:</u>
Alkalinity (as CaCO <sub>3</sub> )	20	mg/L	11/10/2015	13:04	SM 2320 B-97	1	1 3
Nitrogen, Ammonia	<	0.06	mg/L	11/9/2015	12:12	SM 4500-NH3 D-97	1 0.06 0.2
pH - Lab	6.7	SU	11/5/2015	15:40	SM 4500-H+B-96	1	
Residual Chlorine - Lab	<	0.02	mg/L	11/5/2015	10:30	SM 4500-Cl D-93	1 0.02 0.07
Specific Conductance	96	µmhos/cm	11/10/2015	13:57	EPA 120.1	1	1 10
Hardness, Total (as CaCO <sub>3</sub> )	36	mg/L	11/10/2015	9:30	USGS I-1338-85	2	4 10

Results are reported on an as received basis.

< Not detected. Less than LOD.

Report Approved By:

Joseph Dierkes

Project Manager

MN Certification # 027-137-152

Test results in this report relate only to the samples received on the dates indicated. This report must not be reproduced, except in full, without the written approval from Pace Analytical. All tests were performed in-house by Pace Analytical.



# MinnTac

**Pace Analytical**

**Client:** MinnTac

**Pace Project #:** 088594

**Test:** Acute Toxicity Evaluation

**Test Initiation Date:** November 5, 2015

**Test Termination Date:** November 7, 2015

## ENVIRONMENTAL SAMPLE TEST INFORMATION

[illegible]

**TOXICITY TEST RENEWAL FORM**CLIENT: MINNTACPACE PROJECT #: 088594TEST: Acute Toxicity EvaluationTEST INITIATION DATE: November 5, 2015ORGANISM: Fathead MinnowTERMINATION DATE: November 7, 2015

TEST DAY	0 Test Initiation	1	2	3	4
DATE	11/5/15	11/6/15	11/7/15		
TIME OF READING				--	--
				--	--
	FHM 1235	FHM 1215	FHM 1215	FHM	FHM
TIME OF FEEDING	NA	NA	N/A		
DILUTION WATER	RW 088594-2	RW 088594-2	RW —	RW	RW
	MHRW 15-067	MHRW 15-067	MHRW —	MHRW	MHRW
INITIALS	KW	KW	CR		



## INITIAL CHEMISTRIES

CLIENT: MINNTAC

Pace Project #: 088594

TEST: Acute Toxicity Evaluation

TEST INITIATION DATE: November 5, 2015

ORGANISM(S): Fathead Minnow

TEST TERMINATION DATE: November 7, 2015

11/5/15		mw	
CONCENTRATION: MHRW			
pH (su)	7.99		
DO (mg/l)	8.1		
Cond (umhos/cm)	311		
CONCENTRATION: SW002 Receiving Water			
pH (su)	7.23		
DO (mg/l)	8.2		
Cond (umhos/cm)	71		
CONCENTRATION: 100% (WS006/007) 100% Effluent			
pH (su)	7.79		
DO (mg/l)	7.2		
Cond (umhos/cm)	2190		
Daily Temperatures			
25.3			

## FINAL CHEMISTRIES

11/6/15 or 1		11/7/15 or	
CONCENTRATION: MHRW			
pH (su)	8.09	8.13	
DO (mg/l)	8.1	8.1	
CONCENTRATION: SW002 Receiving Water			
pH (su)	7.74	7.86	
DO (mg/l)	8.1	8.0	
CONCENTRATION: 100% (WS006/007) 100% Effluent			
pH (su)	8.33	8.42	
DO (mg/l)	8.0	7.9	
Daily Temperatures			
WB#5 24.4		24.8	

## ACUTE TOXICITY DATA LOG

Client: MINNTAC
Project #: 088594
Test: Acute Toxicity Evaluation
Test Initiation Date: November 5, 2015
Investigator: Dierkes
Test Duration: 48 hour
Renewal: <b>None</b> / Daily / Other

Species: Fathead Minnow
Age: 10 day
No. Animals/No. Reps: 10/2
Sources of Animals: EC&T
Dilution Water/Control: River Water
Test Volume: 600 mL
Test Temperature: 25°C
Minimum Control Survival $\geq 90\%$ ( Y / N )

Concentration	Survival Readings: (# alive out of # exposed from above unless shown otherwise)					
	24 Hour Replicate A      B	48 Hour Replicate A      B	72 Hour Replicate A      B	96 Hour Replicate A      B		
MHRW	10    10	9    10	X	X		
SW002 (RW)	10    10	10    10				
WS006/007 (100% Eff)	10    10	10    10				
	Dated Initials 11/6/15 kw	Dated Initials 11/2/15 or				
Comments:						